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L4: Entry 14 of 14 File: USPT Aug 20, 1991

DOCUMENT-IDENTIFIER: US 5041541 A
TITLE: Functional sugar substituted with reduced calories

Detailed Description Paragraph Right (4):
The term "galactose oxidase" as used herein refers to D-galactose: oxygen 6-oxidoreductase which is identified as E.C. 1.1.3.9 or as Chemical Abstracts Registry Number 9028-79-9.

Detailed Description Paragraph Right (49):
The reaction is conducted in a one liter vessel equipped with an aerator and a gentle stirrer. Sterile conditions are used to prevent enzyme deactivation by microbial contamination. The reaction is run at 4.degree. C. to minimize deactivation of galactose oxidase.

Detailed Description Paragraph Right (50):
Methyl .beta.-D-galactopyranoside (1) is dissolved in the aerated phosphate buffer. The volume flow of air discharged by the aerator is regulated to produce an oxygen saturated solution while preventing foaming of the solution. At 4.degree. C., the galactose oxidase and catalase are added and this solution is aerated for 20 hours.

Detailed Description Paragraph Right (69):
The reaction is conducted in a vessel equipped with a gentle stirrer and an aerator. Sterile conditions are used to prevent enzyme deactivation by microbial contamination. The reaction is run at 4.degree. C. to minimize deactivation of galactose oxidase.

Detailed Description Paragraph Right (70):
Lactitol (23) is dissolved in the aerated phosphate buffer. At 4.degree. C., the galactose oxidase and catalase are added and this solution is aerated to maintain oxygen saturation for 20 hours.

Detailed Description Paragraph Right (73):
The 5-C-hydroxymethylation of galactosyl groups described above is readily adapted by one skilled in the art to other di-, tri- and oligosaccharides containing at least one galactosyl group. Applicable starting compounds for this type of 5-C-hydroxymethylation are raffinose (i.e., 0-.alpha.-D-galacto pyranosyl(1.fwdarw.6)-.alpha.-D-glucopyranosyl-.beta.-D-fructofuranoside), stachyose (i.e., 0-.alpha.-D-galactopyranosyl-(1.fwdarw.6)-0-.alpha.-D-galactopyranosyl-(1. fwdarw.6) -.alpha.-D-glucopyranosyl-.alpha.-D-fructofuranoside), arabino- galactan and D-galactopyranosyl glycerols.

Detailed Description Paragraph Center (9):
1. Oxidation of Methyl .beta.-D-Galactopyranoside with Galactose Oxidase

Detailed Description Paragraph Table (1):
##STR16## Reagents MW Moles Amount
methyl .beta.-D-galactopyranoside 194.18 0.103 20.0 g Sigma Chemical Co., (No. M-6757) Phosphate Buffer, 100 mM -- -- 412.0 ml Catalase, 16900 units/mg -- -- 7.5 mg Sigma Chemical Co., (No. C-40) Galactose Oxidase -- -- 9000 units

Detailed Description Paragraph Table (3):

##STR27## ##STR28## Reagent Amount
Lactitol (23) 20.0 g (manufactured by CCA BioChem) Phosphate Buffer, 100 mM, pH 7 232.0 ml Catalase (Sigma), 16900 u/mg 7.00 mg Galactose Oxidase 9000 units

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L4: Entry 11 of 14 File: USPT Apr 14, 1992

DOCUMENT-IDENTIFIER: US 5104797 A
TITLE: Process for preparing 5-C-hydroxymethyl aldohexose-based compounds

Brief Summary Paragraph Right (17):
Galactose oxidase has the particular characteristic of converting the C-6 hydroxy group in galactose to the corresponding aldehyde (See Mardufu et al., Can. J. Chem., 50, 768 (1971)). The reaction has been successfully applied to a number of mono- and polysaccharides (See Whyte et al., Carbohds. Res., 57, 273 (1977); Jacket et al., Carbohds. Res, 49, 335 (1976)). Root et al., J. Am. Chem. Soc., 107, 2997 (1985), have recently shown that this enzymic synthesis can be applied to polyols. Also, Yalpani and Hall, J. Poly. Sc., 20, 3399-3420 (1982), have cataloged a significant number of applications for the product of the galactose oxidase reaction (e.g., reductive amination, oxidation and reduction).

Detailed Description Paragraph Right (4):
The term "galactose oxidase", as used herein, refers to D-galactose:oxygen 6-oxidoreductase which is identified as E. C. 1.1.3.9 or as Chemical Abstracts Registry Number 9028-79-9.

Detailed Description Paragraph Right (5):
The term "D-aldohexose:oxygen 6-oxidoreductase", as used herein, refers to enzymes which convert the C-6 hydroxy group in an aldohexose to the corresponding aldehyde: ##STR3## One example of a D-aldohexose:oxygen 6-oxidoreductase is galactose oxidase.

Detailed Description Paragraph Right (11):
An aqueous solution having a concentration of from about 1% to about 50%, preferably from about 10% to about 20% of D-aldohexose-based compound is prepared. The pH of the solution is adjusted to enhance reaction kinetics. A solution pH of from about 6 to about 8 is desired when using galactose oxidase as the enzyme. The desired pH may be achieved, for example, by buffering the solution or by simple titration. The solution temperature should be selected so as to minimize enzyme degradation.

Detailed Description Paragraph Right (12):
Galactose oxidase enzymic conversion requires a temperature of from about 1.degree. C. to about 50.degree. C. The reaction can be run at temperatures up to the inactivation temperature of the enzyme. However, at higher temperatures microbial growth can be an issue. A temperature of from about 3.degree. C. to about 25.degree. C. provides good enzyme stability, good oxygen saturation values at standard pressure, and reasonable reaction kinetics for galactose oxidase, and is therefore particularly preferred. Preferably the reaction is run at 3.degree. C. to 6.degree. C. Typical reaction times are in the range of from about 1 to about 24 hours.

Detailed Description Paragraph Right (19):
The preferred reaction utilizes galactose oxidase as the D-alsohexose:oxygen 6-oxidoreductase and a galactose-based starting material. Preferred galactose-based compounds include D-galactose (in all tautomeric forms), D-galactosyl polyols (e.g., lactitol), alkyl D-galactoside (e.g., ethyl galactoside), D-galactitol, D-galactonic acid, and di, tri-, oligo-, or polysaccharides comprising one or more of the above-mentioned simple sugar linkages (e.g., stachyose, raffinose, arabino-galactan). The most preferred reactions utilize about 10 to about 20% D-galactose-based compound solution, a pH from about 5 to about 8, a temperature from about 3.degree. C. to about 6.degree. C., from about 1000 to about 200,000 unit activity galactose oxidase/mole starting material, from about 10,000 to about 2,000,000 unit catalase activity/mole

L4: Entry 13 of 14 File: USPT Oct 1, 1991

DOCUMENT-IDENTIFIER: US 5053225 A
TITLE: Functional organic thin film chemically bonded to biologically active agent

Brief Summary Paragraph Right (17):
As for the polysaccharides, though many compounds can be used, dextran, pullulan, gum arabic, araban, arabogalactan, galactan and starch are representative thereof.

Brief Summary Paragraph Right (46):
oxidoreductase, such as glucose oxidase, aminoacid oxidase, catalase, ascorbate oxidase, xanthene oxidase, cholesterol oxidase, glycerol oxidase, glycerol-3-phosphoric acid oxidase, choline oxidase, acethyl-CoA oxidase, aldehyde oxidase, galactose oxidase, sarcosine oxidase, pyruvate oxidase, lactate oxidase, tyrosinase, peroxidase, etc.;

starting material, from about 0.1 mM to about 2mM CuSO.sub.4, and a reaction time of from about 1 to about 24 hours. hours.

Detailed Description Paragraph Right (22):
Immobilized enzymes are also preferred due to the fact that no subsequent enzyme removal step is required (See Masbach, Methods in Enzymology, Vols. 135 (1987), 136 (1987), 44 (1976)). The use of the triazine-coupling method (See Lily, Methods in Enxymology, Vol. 44, pg. 46 (1976) for the immobilization of galactose oxidase on polyethyleneimine silica (PEI-silica, J. T. Baker) produces effective immobilized enzyme.

Detailed Description Paragraph Right (27):
Another method of conducting the condensation reaction with formaldehyde is through the reaction of the sugar aldehyde which is produced via the galactose oxidase oxidation reaction and formaldehyde on a strongly basic resin. The oxidation product and the formaldehyde are contacted with a resin which has a pH of at least 11.5 at a temperature of from about 20 C. to about 50.degree. C. for from 0.5 to 24 hours. A ratio of formaldehyde to sugar aldehyde of 4:1 to 8:1 is used. Preferably the ratio ia about 4:1 to about 5:1. The resin can have various levels of cross linking, ranging from about 2% to about 8%. Any commercial resin which is strongly basic can be used. Resin usage levels range from stoichiometric amounts (2.9 Meq) to an excess (30 Meq). The amount of resin will control the reaction kinetics of the condensation.

Detailed Description Paragraph Right (65):
The reaction is conducted in a one liter vessel equipped with an aerator and a propeller mixer. The mixer is run at a tip speed of 450 rpm. The reaction is run at 4.degree. C. to minimize deactivation of galactose oxidase.

Detailed Description Paragraph Right (66):
Methyl .beta.-D-galactopyranoside (1) is dissolved in the aerated phosphate buffer containing the dissolved CuSO.sub.4. The volume flow of air discharged by the aerator is regulated to produce an oxygen saturated solution while preventing severe foaming of the solution. A temperature of about 4.degree. C. is maintained. The galactose oxidase and catalase are added and this solution is aerated for 20 hours.

Detailed Description Paragraph Right (70):
The reaction is conducted in a vessel equipped with a gentle aerator and a propeller mixer. The mixer is run at a tip speed of 450 rpm. The reaction is run at 4.degree. C. to minimize deactivation of galactose oxidase.

Detailed Description Paragraph Right (71):
Lactitol (5) is dissolved in the aerated phosphate buffer. At 4.degree. C., the galactose oxidase and catalase are added and this solution is aerated to maintain oxygen saturation for 20 hours.

Detailed Description Paragraph Center (12):
1. Oxidation of Methyl .beta.-D-Galactopyranoside (1) with Galactose Oxidase

Detailed Description Paragraph Type 0 (5):
86,500 unit galactose oxidase activity per mole starting material

Detailed Description Paragraph Table (2):
methyl .beta.-D-galactopyranoside 20.0 g (Sigma Chemical Co., No. M-6757) CuSO.sub.4 66 mg Phosphate Buffer, 100 mM, pH7 412.0 ml Catalase, 16900 126,750 unit activity (Sigma Chemical Co., No. C-40) (approximately 1,231,000 unit activity/ mole starting material) Galactose Oxidase 9,000 unit activity (approximately 87,400 unit activity/mole starting material)

Detailed Description Paragraph Table (4):
##STR8## ##STR9## Reagent Amount
Lactitol (5) 20.0 g (manufactured by CCA BioChem) Phosphate Buffer, 100 mM, pH 7 232.0 ml Catalase (Sigma) 118,300 unit activity (approximately 1,977,000 unit activity/ mole of starting material) Galactose Oxidase 8,280 unit activity (approximately 142,500 unit activity/ mole of starting

material) _____

CLAIMS:

2. A method according to claim 1 wherein said D-aldohexose:oxygen 6-oxidoreductase is galactose oxidase.

5. A method according to claim 3 wherein said galactose oxidase is immobilized.

26. A method according to claim 25 wherein said D-aldohexose:oxygen 6-oxidoreductase is galactose oxidase.

29. A method according to claim 27 wherein said galactose oxidase is immobilized.

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L4: Entry 9 of 14 File: USPT Sep 28, 1993

DOCUMENT-IDENTIFIER: US 5248597 A
TITLE: Method and apparatus for analyzing starch and related carbohydrates

Detailed Description Paragraph Right (32):
Further, the invention is enabled to carry out analyzing of polysaccharide obtained by reacting other monomers, wherein an enzyme electrode for measuring glucose is altered. It is also possible to measure other polysaccharide by the combination of polysaccharide decomposing enzyme and electrode. Taking galactan contained in plants as an example, amount of DE value equivalent (wherein DE value itself is originally defined in regard to starch and the related carbohydrates) of galactan is enabled to be calculated by combining hydrolase which has an activity to cut galactoside linkage and an immobilized galactose oxidase electrode.

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L4: Entry 8 of 14 File: USPT Dec 26, 1995

DOCUMENT-IDENTIFIER: US 5478576 A
TITLE: Arabinogalactan derivatives and uses thereof

Brief Summary Paragraph Right (20):
In one embodiment the antiviral therapeutic agent adenosine arabinoside mono-5'-phosphate (ARA-AMP) is coupled to arabinogalactan. In addition, ARA-A of acyclovir, both antiviral therapeutic agents, may also separately be coupled to arabino-galactans. In another embodiment the radioprotective agent S-2-(3 aminopropylamino) ethylthiophosphoric acid (known as WR2721) is attached to arabinogalactan. The invention provides methods and compositions which enable the attachment of a variety of therapeutic agents to arabinogalactan and the delivery of those agents into the cytoplasm of cells via endocytotic activity of the asialoglycoprotein receptor.

Detailed Description Paragraph Right (1):
The arabinogalactan used here in a preferred embodiment is highly purified and substantially free of endotoxins, and is derived from the Western Larch and has a single peak by size exclusion chromatography of about 20,000 daltons. Arabinogalactan can be used in its native, 20,000 dalton form; alternatively polymers of arabinogalactan (molecular weight greater than the 20,000 dalton form), or degradative products (molecular weight below the 20,000 dalton form) can be used. Purified arabinogalactan has a single peak of 20,000 daltons by gel filtration, and a ratio of galactose to arabinose of 5 to 1 as determined by the alditol acetate method. It binds the asialoglycoprotein receptor on hepatocytes [Josephson Groman et al. Mag. Res. Imag. (1990) 8: 637-646]. It has been shown that L-arabinose, and D-galactose interact with the asialoglycoprotein receptor while, for example, common monosaccharides like glucose or mannose do not [Lee, Haekyung, Kelm, et al., Biol. Chem., Hoppe-Seyler (1988) 369: 705-714]. It has also been shown that an underivatized 4-hydroxy group on galactose and the clustering of suitable sugars, as is displayed by highly branched polysaccharides like arabinogalactan, are important factors in binding the asialoglycoprotein receptor. Given these requirements, and based on the above composition and structure, arabinogalactan is distinguishable from other polysaccharides including dextrans, starches, celluloses, inulins, 1-4 linked galactan and gum arabic. Though chemically distinguishable from arabinogalactan, gum arabic is another polysaccharide which like arabinogalactan interacts with the asialoglycoprotein receptor.

Detailed Description Paragraph Right (6):
In another embodiment, galactose oxidase treatment of arabinogalactan can be used to create aldehyde groups. The aldehyde groups can be reacted with diamino compounds (e.g. ethylenediamine), to form a Schiff base, followed by reduction with sodium borohydride. The resulting amino derivative of arabinogalactan can then be used for the attachment of therapeutic agents.

Detailed Description Paragraph Right (29):
Ten grams of arabinogalactan is dissolved to a total volume of about 50 ml in 0.1M potassium phosphate buffer, pH=6.0. To the resultant solution is added 225 units of galactose oxidase dissolved in about 2 ml of the same buffer. The oxidation is allowed to proceed for 24 hours at room temperature. The H.sub.2 O.sub.2 content is found to be about 3 mg/ml, as measured by peroxide test strips. A second addition of 225 units of GO is made to the reaction mixture. After another 24 hour reaction period the peroxide content is found to be unchanged from the result of the first GO treatment at about 3 mg/ml. Twenty milligrams of catalase (dry solid) is added to decompose the

peroxide. After standing at room temperature overnight the contents of the flask are found to be free of peroxide.

Detailed Description Paragraph Center (12):
Example 10: Treatment of arabinogalactan with galactose oxidase (GO)

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L4: Entry 6 of 14 File: USPT Feb 8, 2000

DOCUMENT-IDENTIFIER: US 6022717 A
TITLE: Use of oxidation promoting chemicals in the oxidation oxidizable galactose type of alcohol configuration containing polymer

Abstract Paragraph Left (1):
Process for the oxidation of the oxidizable galactose type of alcohol in oxidizable galactose type of alcohol configuration containing polymer, such as guar, with galactose oxidase in the presence of oxidation promoting chemicals.

Brief Summary Paragraph Right (2):
The present invention relates to the oxidation of oxidizable galactose type of alcohol configuration containing polymer and more particularly it relates to the use of oxidation promoting chemicals in such oxidation by galactose oxidase.

Brief Summary Paragraph Right (4):
The product of the oxidation of aqueous solutions of guar gum and other galactose bearing polysaccharides using galactose oxidase enzyme was disclosed by F. J. Germino in U.S. Pat. No. 3,297,604. The aldehyde bearing oxidized products are separated by precipitation from the aqueous solutions used for the enzyme reactions. Germino disclosed the use of the oxidized products in the manufacture of paper. The aldehyde bearing oxidized products were disclosed to be also suitable for use to crosslink polyamino polymers, polyhdroxy polymers, and proteins.

Brief Summary Paragraph Right (5):
C. W. Chiu, et.al., U.S. Pat. No. 5,554,745, discloses (1) the preparation of cationic galactose containing polysaccharides and (2) the enzymatic oxidation in aqueous solution of the cationic galactose containing polysaccharides with galactose oxidase. The oxidized cationic polysaccharides are disclosed to improve the strength characteristics of paper.

Brief Summary Paragraph Right (6):
According to the present invention there is provided a process for the oxidation of the oxidizable galactose type of alcohol configuration to aldehyde in oxidizable galactose type of alcohol configuration containing polymers comprising providing oxidizable galactose type of alcohol configuration containing polymer and galactose oxidase and oxidation promoting chemical and contacting them.

Brief Summary Paragraph Right (7):
It has surprisingly been discovered that the use of oxidation promoting chemicals, e.g., 1,2-benzisothiazolin-3-one, in the oxidation of oxidizable galactose type of alcohol configuration containing polymers, e.g., guar, by galactose oxidase, results in increased levels of oxidation and corresponding increase in paper strength characteristics when the oxidized galactose type of alcohol configuration containing polymer is employed in the papermaking process.

Brief Summary Paragraph Right (10):
The oxidizable galactose type of alcohol configuration containing polymers can be galactomannan gums or their ether derivatives, arabinogalactan gums or their ether derivatives, other gums or their ether derivatives, galactoglucomannan hemicelluloses or their ether derivatives and synthetically or enzymatically modified polymers. Preferred galactomannan gums are guar, locust bean, tara and fenugreek. Preferred arabinogalactan gums are arabic, larch and tragacanth gums. Preferred synthetically of enzymatically modified polymers are galactose deficient polysaccharides,

polyacrylamides, polyacrylates, polyamides, polyvinyl alcohol, and polyvinyl acetate. Most preferred such polymers are starch and polyacrylates. The phrase "galactose deficient" as used in the present application means that the oxidizable galactose type of alcohol configuration containing polymer it refers to contains less than 20% of oxidizable galactose type alcohol configuration based on the weight of the oxidizable galactose type of alcohol configuration containing polymer. Preferred other gums are carubin, lichenan, tamarind and potato galactan. Most preferred oxidizable galactose type of alcohol configuration containing polymers are guar gum and its ether derivatives such as cationic anionic, amphoteric, hydroxypropyl, dihydroxypropyl and hydroxyethyl guar.

Brief Summary Paragraph Right (14):
Galactose oxidase (EC 1.1.3.9) is a copper oxidase which converts the oxidizable galactose type of alcohol configuration to the corresponding aldehyde group (thus producing oxidized galactose) by reducing oxygen to hydrogen peroxide. The copper must be in the correct oxidation state (Cu.sup.2+) to perform this oxidation and the copper ion must be retained in the galactose oxidase. If the galactose oxidase solution is stored anaerobically with any oxidizable substrate, it can become inactive. Galactose oxidase can be reactivated by oxidizing the copper with reagents such as potassium ferricyanide. Another way to oxidize the copper in galactose oxidase would be by electrochemical oxidation.

Brief Summary Paragraph Right (15):
Galactose oxidase can be obtained by any suitable manner, e.g., by fermenting various wild type and cloned fungi but is usually obtained from Fusarium spp (NRRL 2903). Cultures may also be obtained from the American Type Culture Collection under Dactylium dendroides ATCC 46032 and they are successfully fermented under the procedure of Tressel and Kosman. Methods in Enzymology, Vol 89 (1982), pg 163-172. The gene for active forms of the enzyme have been expressed in E. coli and Aspergillus and this development may lead to more stable and active forms of the enzyme as well as much greater production levels. The gene or improved forms will also be expressed in plants which can be harvested to give higher levels of enzyme without the threat of enzyme destruction by proteases in a fermentation broth.

Brief Summary Paragraph Right (17):
The treatment of oxidizable galactose type of alcohol configuration containing polymer with galactose oxidase and catalase is the subject of companion application Ser. No. 09/801,789 filed on Dec. 31, 1997 (Hercules Docket No. PCH 5484, "Oxidation in Solid State of Oxidizable Galactose Type of Alcohol Configuration Containing Polymers" by R. L. Brady, R. T. Leibfried and T. T. Nguyen), the disclosure of which is hereby incorporated by reference.

Brief Summary Paragraph Right (18):
Preferably the oxidation of oxidizable galactose type of alcohol configuration containing polymer with galactose oxidase is carried out in the presence of means to decompose the hydrogen peroxide generated during the conversion of the oxidizable galactose type of alcohol configuration to aldehyde. Preferably the means to decompose hydrogen peroxide is catalase.

Brief Summary Paragraph Right (20):
The oxidizable galactose type of alcohol configuration containing polymer can be oxidized in solid form, in slurry form or in solution. The oxidation can be carried out enzymatically by galactose oxidase. Preferably neutral, cationic or anionic or amphoteric guar that has been oxidized by galactose oxidase and catalase is used in the present invention. Galactose oxidase can be applied to solid, slurry, or solution forms of guar products: e.g., shredded, powder, flake, and pellet forms of neutral, cationic, anionic or amphoteric guar. Guar derivatives, such as those containing hydroxypropyl groups can also be used.

Brief Summary Paragraph Right (22):
When the oxidizable galactose type of alcohol configuration containing polymer is contacted with galactose oxidase in aqueous medium the lower limit of the oxidizable galactose type of alcohol configuration containing polymer can be about 0.001%, preferably about 0.2% and most preferably about 8%. In this instance the upper limit of the oxidizable galactose type of alcohol configuration containing polymer can be

about 50%, preferably about 30% and most preferably about 20%, all based upon the weight of the composition.

Brief Summary Paragraph Right (23):
When solid oxidizable galactose type of alcohol configuration containing polymer is contacted with solid galactose oxidase, the lower limit of the oxidizable galactose type of alcohol configuration can be about 50% based upon the weight of the composition. Preferably the lower limit is about 70% and most preferably it is about 85%. When solid oxidizable galactose type of alcohol configuration containing polymer is contacted with solid galactose oxidase the upper limit of the oxidizable galactose type of alcohol configuration containing polymer can be about 100% based upon the weight of the composition. Preferably it can be about 98% and most preferably about 95%.

Brief Summary Paragraph Right (24):
An International Unit (IU) of galactose oxidase will convert one microequivalent of the oxidizable galactose type of alcohol configuration containing polymer to aldehyde per minute at 25.degree. C. and pH 7.0. The unit can be measured by coupled assays where the by-product H.sub.2 O.sub.2 is used by peroxidases to oxidize dye precursors, giving a chromophore. The production of the chromophore is measured by light absorbance at a wavelength suitable to the dye used (o-tolidine, 425 nm; o-dianisidine, 436 nm; 2,2.sup.1 -azinobis(3-ethylbenzo-thiazoline-6-sulfonic acid), diammonium salt (ABTS), 405 nm). The method using the ABTS dye is used to determine International Units (IU).

Brief Summary Paragraph Right (25):
The lower limit of the galactose oxidase can be about 10 IU per gram of oxidizable galactose type of alcohol configuration containing polymer. Preferably the lower limit is about 25 and most preferably about 35 IU per gram of oxidizable galactose type of alcohol configuration containing polymer. The upper limit of the galactose oxidase can be about 3,000 IU per gram of oxidizable galactose type of alcohol configuration containing polymer, preferably about 2,000 and most preferably about 1,000 IU per gram of oxidizable galactose type of alcohol configuration containing polymer.

Brief Summary Paragraph Right (26):
The lower limit of catalase can be about 1, preferably about 50 and most preferably about 100 IU of catalase/IU of galactose oxidase. The upper limit of catalase can be about 10,000, preferably about 5,000 and most preferably about 1,000 IU of calatase/IU of galactose oxidase. One (1) IU of catalase will convert a micromole (10.sup.-6 mole) of hydrogen peroxide to water and oxygen per minute at pH 7.0 and 25.degree. C.

Detailed Description Paragraph Right (1):
This example shows the effect of various oxidation promoting chemicals on the oxidation of shredded guar. Shredded guar was oxidized at 0.2% in deionized water by adding 1% of various oxidation promoting chemicals, 540 IU of galactose oxidase (Sigma G7400)/g of guar and 1852 IU of catalase (Sigma C40)/IU of galactose oxidase. The resulting solutions were stirred for 3 days at room temperature. Table 1 shows the oxidase promoting chemicals and the results of the iodometric titration for aldehyde at the end of 3 days. Theoretical full reaction would give 2.06 meq/g for the aldehyde. All the oxidation promoting chemicals aid the oxidation so that a higher level of aldehyde content is obtained.

Detailed Description Paragraph Right (2):
This example shows the effect of higher levels of Proxel GXL on the oxidation of guar. To a 0.2% aqueous solution of Supercol U neutral guar powder was added 1% or 10% (based on guar) of 1,2-benzisothiazolin-3-one. Catalase (Sigma C40) was added at 1852 IU/IU of galactose oxidase. Galactose oxidase was added at 108 IU/g of guar.

Detailed Description Paragraph Right (4):
This example shows the improvement in paper strength that can be attained by using an oxidation promoting chemical in the oxidation process. Neutral shredded guar was used at 0.2% in water. Proxel GXL was added as indicated to give a level of 1,2-benzisothiazolin-3-one of 1% based on the guar. Catalase (Sigma C40) at 1852 IU/IU of galactose oxidase (Sigma G7400) at 540 IU/g guar were added to the solutions. Samples were mixed overnight before titration and papermaking. Handsheets were made at

80 lb/3000 ft.sup.2 basis with bleached kraft pulp and an oxidized guar level of 1% based on dry weight of the pulp. Table III shows the results for aldehyde level (iodometric titration) and paper dry tensile strength for oxidized guar with and without Proxel GXL. Using Proxel GXL resulted in a much higher oxidation level and greatly improved paper properties.

Other Reference Publication (1):
Derwent Abstract WPIL 88-258460/37 EP-281655 "Starch Ether Derivs.-Used in Prepn of Aldehyde-Contg. Hetero:Polysaccharide(s), by Reacting with Galactose Oxidase", Sep. 14, 1988.

CLAIMS:

1. A process for the oxidation of an oxidizable galactose type of alcohol configuration to aldehyde in an oxidizable galactose type of alcohol configuration containing polymer comprising providing the oxidizable galactose type of alcohol configuration containing polymer and galactose oxidase and oxidation promoting chemical and contacting them, wherein the oxidizable galactose alcohol type of configuration is described by the following chemical structures ##STR2## where, R1 is an alkyl group of the formula C(n)H(2n+1)where n is 0 to 20; z is 0 or 1; where R2 is a linking group composed of an alkylene, or an aromatic alkylene, or an alkylene ether, or an alkylene ester, or an alkylene amide, or an alkylene urethane diradical where said linking groups has a total number of carbon from 2 to 20; where R3 is --H, --OH, --OCH.sub.3, --OC.sub.2 H.sub.5, --OC.sub.3 H.sub.7, --OOCR5, (where R5 is alkyl radical of 1 to 5 carbons), --NH.sub.2, --NH--CO--R5; and y is 0 or 1; and wherein the oxidizable galactose type of alcohol configuration containing polymer is selected from the group consisting of galactomannan gums or their ether derivatives, arabinogalactan gums or their ether derivatives, other gums or their ether derivatives, galactoglucomannan hemicelluloses or their ether derivatives and galactose deficient polysaccharides, polyacrylamides, polyacrylates, polyamides, polyvinyl alcohol, and polyvinyl acetate.

4. The process of claim 1 wherein the lower limit of oxidizable galactose type of alcohol configuration containing polymer is about 0.001% based on the total weight of oxidizable galactose type of alcohol configuration containing polymers, galactose oxidase and oxidation promoting chemical and the lower limit of galactose oxidase is about 10 IU/g of oxidizable galactose type of alcohol configuration containing polymers and the lower limit of oxidation promoting chemical is about 0.1% based on the total weight of oxidizable galactose type of alcohol configuration containing polymers, galactose oxidase and oxidation promoting chemical.

5. The process of claim 1 wherein the upper limit of oxidizable galactose type of alcohol configuration containing polymer is about 99% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical, the upper limit of galactose oxidase is about 3,000 IU/g of oxidizable galactose type of alcohol configuration containing polymer and the upper limit of oxidation promoting chemical is about 5% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical.

8. The process of claim 6 wherein the means to decompose hydrogen peroxide is catalase in an amount of at least about 1 IU of catalase per unit of galactose oxidase.

9. The process of claim 6 wherein the means to decompose hydrogen peroxide is catalase in an amount of up to about 10,000 IU of catalase per unit of galactose oxidase.

10. The process of claim 2 wherein the oxidizable galactose type of alcohol configuration containing polymer is selected from the group consisting of galactomannan gums or their ether derivatives, arabinogalactan gums or their ether derivatives, other gums or their ether derivatives, galactoglucomannan hemicelluloses or their ether derivates and synthetically or enzymatically modified polymer, the lower limit of oxidizable galactose type of alcohol configuration containing polymer is about 0.001% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical, the lower limit of galactose oxidase is about 10 IU/g of oxidizable galactose type of

alcohol configuration containing polymer and the lower limit of oxidation promoting chemical is about 0.1% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical, the upper limit of oxidizable galactose type of alcohol configuration containing polymer is about 99% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical, the upper limit of galactose oxidase is about 3,000 IU/g of oxidizable galactose type of alcohol configuration containing polymer and the upper limit of oxidation promoting chemical is about 5% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical, means to decompose hydrogen peroxide is added and the means to decompose hydrogen peroxide is catalase in an amount of at least about 1 IU per IU of galactose oxidase and up to about 10,000 IU per unit of galactose oxidase.

12. The process of claim 10 wherein the galactomannan gum is selected from the group consisting of guar, locust bean, tara and fenugreek gum or their ether derivatives; the arabinogalactan gum is selected from the group consisting of arabic, larch and tragacanth gum or their ether derivatives, the other gum is selected from the group consisting of carubin, lichenan and potato galactan gum or their ether derivatives and the synthetically or enzymatically modified polymer is selected from the group consisting of galactose deficient polysaccharides, polyacrylates, polyacrylamides, polyvinyl alcohol and polyvinyl acetate.

13. The process of claim 10 wherein the lower limit of oxidizable galactose type of alcohol configuration containing polymer is about 0.2% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical, the lower limit of galactose oxidase is about 25 IU/g of oxidizable galactose type of alcohol configuration containing polymer, the lower limit of oxidation promoting chemical is about 0.5% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical and the lower limit of catalase is about 50 IU per unit of galactose oxidase.

14. The process of claim 10 wherein the upper limit of oxidizable galactose type of alcohol configuration containing polymer is about 98% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical, the upper limit of galactose oxidase is about 2,000 IU/g of oxidizable galactose type of alcohol configuration containing polymer, the upper limit of oxidation promoting chemical is about 3% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical and the upper limit of catalase is about 5,000 IU per IU of galactose oxidase.

17. The process of claim 10 wherein the lower limit of oxidizable galactose type of alcohol configuration containing polymer is about 8% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical, the lower limit of galactose oxidase is about 35 IU/g of oxidizable galactose type of alcohol configuration containing polymer, and the lower limit of oxidation promoting chemical is about 1% based on the total weight of guar, galactose oxidase and oxidation promoting chemical and the lower limit of catalase is about 100 IU per IU of galactose oxidase.

18. The process of claim 10 wherein the upper limit of oxidizable galactose type of alcohol configuration containing polymer is about 95% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical, the upper limit of galactose oxidase is about 1000 IU/g of oxidizable galactose type of alcohol configuration containing polymer, the upper limit of oxidation promoting chemical is about 2% based on the total weight of oxidizable galactose type of alcohol configuration containing polymer, galactose oxidase and oxidation promoting chemical and the upper limit of catalase is about 1,000 IU per IU of galactose oxidase.

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L4: Entry 2 of 14 File: USPT Dec 18, 2001

DOCUMENT-IDENTIFIER: US 6331619 B1
TITLE: Superabsorbent material and method for producing said material

Brief Summary Paragraph Right (7):
The polysaccharides to be used according to the present invention are in particular .alpha.-glucans like starch, amylose and amylopectin, .beta.-glucans like cellulose, galactomannans like guar gum (guaran) and locust bean gum, glucomannans including e.g. xanthan gum, fructans, (arabino)xylans, galactans including alginates and pectins, as well as non-ionic derivatives such as hydroxyethyl and hydroxypropyl derivatives of such polysaccharides. Starch and guar, and to a somewhat lesser extent, cellulose, are preferred for economic reasons. The chain of the polysaccharides is important although there is no critical minimum for the molecular weight. In general, polysaccharides having a molecular weight of more than 1,000 are preferred. A molecular weight above about 25,000 may have a positive effect on the properties of the oxidised product.

Brief Summary Paragraph Right (11):
As an example, the oxidation of starch with nitrite and nitrate in phosphoric acid, mainly resulting in 6-carboxy starch, is described in NL patent application 9301172. An improved oxidation of the 6-hydroxyl groups in starch, using a hypohalite in the presence of a di-tert-alkynitroxyl catalyst is disclosed in WO 95/07303. Examples of oxidation of glucans at the C2-C3 function include the process according to EP-A427349, using low levels of hypobromite, and the process according to WO 94/21690, which uses hydrogen peroxide in the presence of alkali metal; or transition metals. WO 95/12619 describes an improved oxidation of starch with periodic acid, resulting in dialdehyde starch with extensive regeneration of a periodic acid. The dialdehyde starch can be further oxidised to dicarboxyl starch using e.g sodium chlorite and/or hydrogen peroxide. Also, dialdehyde starch can be further oxidised with iodine or bromine or with nitrogen dioxide producing dicarboxy or up to tricarboxy starch. Other known oxidation methods include metal-catalysed oxidation, eg, using ruthenium, anhydrous oxidation using nitrogen dioxide in e.g. halocarbons and enzymatic and chemo-enzymatic oxidation of starch, guar and other polysaccharides, and these can also be used in the present invention. In case of galactomannans, which have terminal galactose units, enzymatic oxidation using galactose oxidase (EC 1.1.3.9) can also be used to introduce aldehyde groups, which can easily be converted to carboxyl groups, e.g. using hypoiodite.